PROCEEDINGS

53. IWK

Internationales Wissenschaftliches Kolloquium
International Scientific Colloquium

Faculty of
Mechanical Engineering

PROSPECTS IN MECHANICAL ENGINEERING

8 - 12 September 2008

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TECHNISCHE UNIVERSITÄT
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Published by
Impressum

Publisher Herausgeber	Der Rektor der Technischen Universität Ilmenau Univ.-Prof. Dr. rer. nat. habil. Dr. h. c. Prof. h. c. Peter Scharff
Editor Redaktion	Referat Marketing und Studentische Angelegenheiten Andrea Schneider Fakultät für Maschinenbau Univ.-Prof. Dr.-Ing. habil. Peter Kurz, Univ.-Prof. Dr.-Ing. habil. Rainer Grünwald, Univ.-Prof. Dr.-Ing. habil. Prof. h. c. Dr. h. c. mult. Gerd Jäger, Dr.-Ing Beate Schlütter, Dipl.-Ing. Silke Stauche
Editorial Deadline Redaktionsschluss	17. August 2008
Publishing House Verlag	Verlag ISLE, Betriebsstätte des ISLE e.V. Werner-von-Siemens-Str. 16, 98693 Ilmenau

CD-ROM-Version:

Implementation Realisierung	Technische Universität Ilmenau Christian Weigel, Helge Drumm
Production Herstellung	CDA Datenträger Albrechts GmbH, 98529 Suhl/Albrechts

ISBN: 978-3-938843-40-6 (CD-ROM-Version)

Online-Version:

Implementation Realisierung	Universitätsbibliothek Ilmenau ilmedia Postfach 10 05 65 98684 Ilmenau

Home / Index:
http://www.db-thueringen.de/servlets/DocumentServlet?id=17534

53rd Internationales Wissenschaftliches Kolloquium
Technische Universität Ilmenau
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Hydrothermal Synthesis of Multiferroic $BiFeO_3$ Fine Particles by La Substitution

NANO MATERIALS AND PROCESSING

Recently, much attention has been focused on developing multiferroic materials, in which the properties of ferroelectricity, ferromagnetism, and ferroelasticity are combined in a single compound. Such materials can be used to develop new applications, including spintronics, data storage media, multiple-state memories, transducers, actuators, etc. [1,2]. $BiFeO_3$ is the most interesting candidate of the few multiferroics having an antiferromagnetic behavior with a relatively high Néel temperature (T_N~370°C) and a ferroelectric behavior with a high Curie temperature (T_C~810°C) and has a rhombohedrally distorted perovskite structure [3]. Though $BiFeO_3$ was discovered in the 1960s, the scarcities, such as presence of impurity phases, high leakage current, low resistivity, small spontaneous polarization, inhomogeneous magnetic spin structure, ferroelectric reliability and high defect density, have hampered its wide practical application. Intensive efforts have been devoted to overcome these obstacles by appropriate cationic substitutions using various techniques. However, it is fascinating to find that no report on synthesis of La-modified $BiFeO_3$ by a low temperature hydrothermal method has been published. It is therefore of interest to synthesis $BiFeO_3$ by La substitution under hydrothermal conditions. In this abstract, we report our experimental study on the preparation and characterization of La-doped $BiFeO_3$ prepared by hydrothermal processing.

$Bi_{1-x}La_xFeO_3$ for x=0.05, x=0.10 and x=0.15 were synthesized by a hydrothermal method. As the starting materials, $Bi(NO_3)_3{\cdot}5H_2O$, $Fe(NO_3)_3{\cdot}9H_2O$ and $La(NO_3)_3{\cdot}6H_2O$ with 99%+ purity were carefully weighted in stoichiometric proportion. A mixture was treated in a 40 ml of KOH as well known mineralizer. The mixture was thoroughly stirred and then dispersed for 15 min. The suspension was transferred into a 40 ml Teflon-lined autoclave with a filling capacity 75%. The hydrothermal treatment was subsequently carried out for the different concentrations of the

mineralizer (2-8 M), reaction temperatures (160-200°C) at reaction time of 16 h under autogenous pressure. After the autoclave was cooled down naturally to room temperature, the final products in the autoclave was filtered out, washed with distilled water and absolute ethanol several times and dried at 80°C for 5 h. The crystal structure of the powders was determined by an X-ray diffraction (Dmax-3C, Rigaku, Japan) using CuKα radiation (λ =1.5406 Å). The microstructures of the samples were examined by a scanning electron microscope (JSM-5310, JEOL, Japan). Chemical analysis of the powders was done using energy dispersive X-ray analysis (EDAX). The vibrating sample magnetometer, LakeShore VSM 7307 (Lake Shore Cyrotronics, Westerville, OH), was used to measure the magnetic hysteresis loop of the powders.

The XRD patterns of the synthesized samples as a function of La content are shown in Fig.1. The diffraction patterns of as-received samples agreed well and indexed to a $BiFeO_3$ with a rhombohedrally distorted perovskite structure belonging to the *R3c* space group (JCPDS Card No. 86-1518). No impurity phases could be detected. This implies that a pure phase $Bi_{1-x}La_xFeO_3$ crystals could be obtained under the present hydrothermal conditions at 200°C for 16 h using the KOH concentration of 4 M. Increase in the La content leads to a decrease in the unit cell volume with a significant decrease in both *a* and *c* parameters in the c/a ratio from 2.490 for *x*=0.0 to 2.483 for *x*=0.15. The calculated lattice parameters (Table 1) are compatible with the reported data within the error range.

Table 1. Lattice parameters and unit cell volumes of the synthesized $Bi_{1-x}La_xFeO_3$

x	a=b	c	c/a	V
0.00	5.583	13.906	2.490	375.46
0.05	5.580	13.882	2.487	374.61
0.10	5.576	13.861	2.485	373.32
0.15	5.574	13.843	2.483	372.50

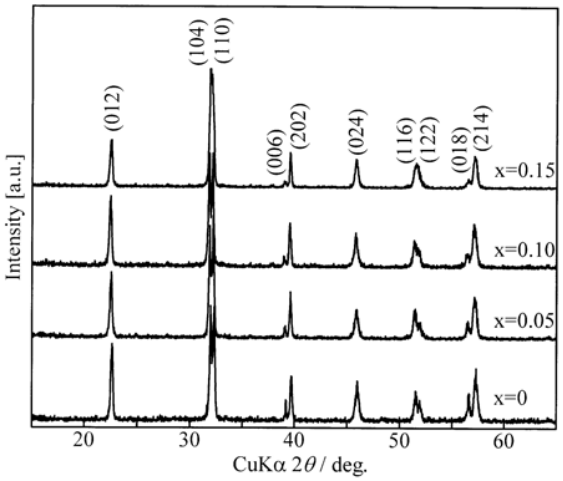

Fig.1. XRD patterns of $Bi_{1-x}La_xFeO_3$ powders synthesized at 200°C for 16 h using the KOH concentration of 4 M.

Fig.2 displays SEM images of pure and La-modified $BiFeO_3$ powders. The grain morphology and the particle size change depended on the substituted La content. SEM images reveal that particles are nearly regular in the submicron range. The particle size increases with increasing La content and it varies in the range 0.3-1 μm.

Fig.3 compares M-H curve of as-received samples as a function of La content. The

substitution of La for Bi suppresses the spin magnetic structure and gives a weak ferromagnetism at room temperature. The magnetism of (BiLa)FeO_3 increases with the content of La due to increscent suppressing spiral magnetic structure and magnetic contribution of La [4].

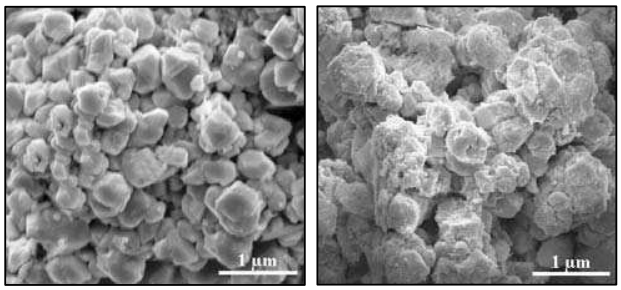

Fig.2. SEM images of $BiFeO_3$ (left) and $Bi_{0.85}La_{0.15}FeO_3$ (right) powders synthesized at 200°C for 16 using the KOH concentration of 4 M.

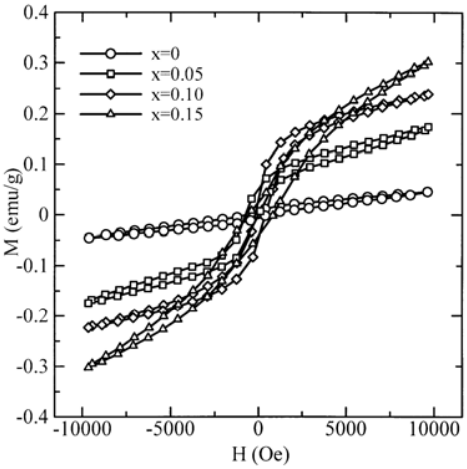

Fig.3. *M-H* curve measured at room temperature for the $Bi_{1-x}La_xFeO_3$ synthesized by the hydrothermal process at 200°C for 16 h using the KOH concentration of 4 M.

The formation process can be described as follows: first, $Bi(NO_3)_3$, $Fe(NO_3)_3$ and $La(NO_3)_3$ transformed into hydroxides, then the $Fe(OH)_3$, *(1-x)*$Bi(OH)_3$ and *x*$La(OH)_3$ were reacted to form the final products of $Bi_{1-x}La_xFeO_3$.

In summary, La-modified $BiFeO_3$ fine particles have been synthesized by a simple hydrothermal process. The replacement of Bi by La had a great effect on the structure, morphology and magnetic properties consequently. Observed magnetic loops of the as-received samples at RT were similar to the previous reported data. The 'dissolution – nucleation – crystallization' process can be used to express crystal growth mechanism of $Bi_{1-x}La_xFeO_3$ under mild hydrothermal treatment.

Acknowledgement:
This work was supported by Program for New Century Excellent Talents in University (NCET) from the Ministry of Education of China.

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